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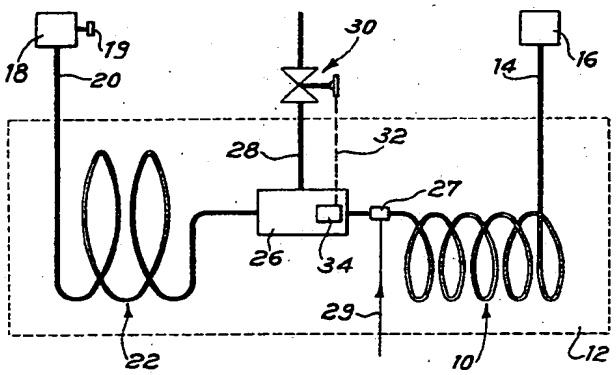
(54) A device for vaporization injection in a gas-chromatographic column.

(57) This invention relates to a device for vaporization injection in a gas chromatographic column housed in an oven. In order to avoid the drawbacks and problems arising with known vaporization injection techniques above all with samples with solvents, the device comprises a direct on column injector suitable for injecting the liquid sample into the initial zone of a capillary column and capable of pneumatically sealing the injection port at the end of injection: between the injector and the gas chromatographic column itself a capillary column length is provided in which vaporization of the injected sample occurs by heating an oven housing said capillary column length, said oven being the same or separate from the oven containing said gas chromatographic column.



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TITLE

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This invention relates to a device to perform vaporization injections in a gas chromatographic column, which is housed in an oven, subject to a preset temperature program to allow a chromatographic separation of the components of an injected sample, as well as the transfer of said components to a detector placed downstream the column, usually outside the oven.

In case of vaporization injection in gas chromatographic columns, so-called vaporization injectors are used, in which the liquid sample is vaporized by the high temperature of a chamber appertaining to the injectior itself and wherein the sample is injected. After vaporization, the sample is sent to the gas chromatographic column by a carrier gas. However, the known vaporization injectors present many drawbacks, specially when the sample contains compounds having different volatility (volatile and high-boiling together) and thermolabile components.
Other drawbacks of known vaporization injectors are due to the need of having a suitably heated vaporization chamber and means to close said chamber on the sample injection side (in particular comprising a duct for an injection syringe needle) both during injection and at the end of same, and this above all when samples including a solvent, which has the tendency to quickly vaporize, are treated.

Accordingly, an object of this invention is to provide a new device wherein the problems and drawbacks of known va-

porization injectors are completely avoided and wherein a perfect vaporization injection, with a consequently perfect analysis of the injected samples, are ensured. Essentially, according to the invention, said device is characterized in that it comprises a direct on column injector, of the type suitable for directly injecting a liquid sample into the initial part of a capillary column, as well as section of capillary column placed upstream the gas chromatographic column and fed by said direct on-column injector, said section of capillary column being housed in an oven for heating and vaporizing the injected sample.

In this way, the injection is carried out with a liquid sample, which maintains its liquid condition during the whole injection stage inside the injector itself and in the initial part of said section of capillary column, and is vaporized only later, at the end of injection, when the oven is heated to cause vaporization and the subsequent beginning of separation of the sample components inside said section of capillary column.

Alternately, the oven can be kept in temperature, and in this case cooling of the injector and of the initial section of capillary column is carried out to keep the sample in liquid condition.

When a splitting operation has to be foreseen, that is an outlet discharge of part of the injected sample after vaporization thereof, so as to send only part of injected sample into the gas chromatographic column, said splitting is carried-out between said section of capillary column and the gas chromatographic column, the splitting

rate being controlled by suitable valves which are operated by a sensor detecting the oven temperature, in order to maintain at a constant value said splitting ratio, namely the ratio between the sample flow sent to the gas chromatographic column and the sample flow upstream the splitting point, while temperature varies. Both in the previous case and in the case wherein said section of capillary column designed for sample vaporization is housed in a separate oven which is independently controlled with respect to the oven of the gas chromatographic column, it is advisable that, in the splitting zone between the two columns and upstream the splitting point, an additional flow of carrier gas is fed through a suitable line (make-up line), in order to obtain a mixing as homogeneous as possible of said carrier with the flow coming out of the vaporization column. In fact, thanks to said additional carrier flow, which can be adjusted, it is possible to control the time the sample remains inside the vaporization capillary column independently from the splitting ratio, while without said make-up line the stay time of sample in the vaporization column would depend from the splitting ratio and would result the lowest the highest the splitting ratio is. Furthermore, a suitable regulation of the flowrate in the make-up line to obtain the optimum sample stay time, allows to reach a further advantage, in that a relatively reduced band amplitude of the sample is achieved, both in the vaporization capillary column section and in the gas chromatographic column. If, despite this, the band amplitude of the sample is still too

wide, it is advisable to insert, upstream the chromatographic column, a trap carrying out a sample re-concentration.

Figure 1 is a diagrammatic view of a first embodiment of the invention.
Figure 2 is a diagrammatic view of a second embodiment, with separate ovens.
Figure 3 is a diagrammatic view of means conveving additional carrier gas and of splitting means.

Referring to figure 1, a gas chromatographic column 10 is housed in an oven 12 and the outlet 14 of same column is connected to a detector 16 for analysis of the injected samples. The samples (comprising their solvents) are introduced by means of a direct on column injector 18 (which is known per se) positioned outside the oven and having a valve 19 acting on a duct in which the needle of an injection syringe is inserted, said needle injecting the liquid sample into the initial part of a capillary column 20, which develops, as indicated by reference 22, inside the oven 12. The capillary column 22 and the gas chromatographic column 10 are connected between them by a splitting device 26 having splitting duct 28 for discharging a given amount of the sample before analysis.
Reference 27 indicates a so-called cool "trap", which is cooled through a line 29, appertaining to the column 10 itself or in the form of an indepentent element placed between the column and splitting device 26. The splitting flow, which is usually a very high percentage of the sample injected into column, is controlled by means

of a valve device 30, which, on its turn, can be advantageously controlled, through a connection 32, by a detector 34 of the oven temperature, in order to maintain a constant splitting ratio notwithstanding temperature variations.

To perform a gas chromatograpnic analysis in the case of the embodiment of fig. 1, the valve 19 of injector 18 is opened and the liquid sample is injected into the initial length of the capillary column 22. No vaporization occurs because the oven 12 is kept cold, or because the injector and the fist length of column are cooled (secondary cooling). Once the injection is carried out and the valve 19 is closed, vaporization is performed or completed.

After vaporization, splitting takes place at 26 with a splitting ratio depending on the concentration and other features of the sample. The trap 27 can be actuated, in case it is necessary or advisable to decrease the band amplitude of sample before it enters the gas chromatographic column.

Another embodiment of the invention is illustrated in figure 2, where the same components as in fig. 1 are indicated by the same reference numerals. This embodiment differs from the previous one in that the capillary column 22 is housed in its own oven 36, independent from the oven 12 of the gas chromatographic column 10, said oven 36 being controlled independently from oven 12.

In correspondence to the splitting zone 26, between said two ovens 12 and 36, a so called make-up line 38 is also provided, which controls the introduction of a further flow of carrier gas through a flow regulating valve, immediately upstream the splitting point. A similar make-up

line can also be provided in the splitting device 26 of figure 1.

This carrier feeding can be performed in any way, provided that, before the splitting drawing, the entering carrier be mixed as homogeneously as possible with the flow coming out from the vaporization column.

This is in particular obtained with the embodiment of fig. 3, where a venturi-shaped tube 42 is used, in the narrow zone 44 of which a mixing of the injected and vaporized sample coming from column 22 and of the carrier flow of make-up line 38 is carried out. Downstream said section 44, the sample is partly fed to the chromatographic column 10 and partly discharged into the splitting line 28.

As mentioned, said make-up line allows to obtain, by a regulation of the carrier flowing through valve 40, an optimum stay time of sample in the column 22, in relation to the sample nature (specially its volatility) and the temperature of oven 36, and this independently from the splitting ratio, which can be separately regulated (by means of valve 30), on the basis of other sample features.

It must be noticed that as the capillary column 22 can perform sample vaporization even gradually, the splitting operation in this case, as well as in the case of the previously described embodiment, goes on during the whole analysis and is not limited to the sample injection stage, as, on the contrary, it usually occurs when conventional vaporization injectors are used.

The shown embodiments can undergo to several modifications, without departing from the spirit and scope of the present invention.

CLAIMS

1.- A device for vaporization injection in a gas chromatographic column housed in an oven, and comprising a direct on column injector for injecting a liquid sample into a initial zone of a capillary column, as well as a capillary column length placed upstream the gas chromatographic column and fed by said direct on column injector, said capillary column length being housed in an oven for heating and vaporizing the injected sample.

2.- A device according to claim 1, wherein between said capillary column length and said gas chromatographic column, a splitting line for discharging a part of the vaporized sample is provided for, said splitting line being equipped with valve means to control the discharged sample flow.

3.- A device according to claim 2, wherein said splitting control valve means are in turn controlled in relation to the oven temperature to obtain a constant splitting ratio (the flow sent to the chromatographic column versus the flow fed upstream the splitting point).

4.- A device according to claim 1, 2 or 3, wherein said capillary column length is housed in a separate oven which is submitted to a temperature variation program independent from the one of the oven containing said gas chromatographic column, said capillary column length

being connected to said gas chromatographic column through a fitting comprising a splitting branching.

5.- A device according to claim 2 or 4, wherein said fitting is fed, upstream the splitting point, with an adjustable flow of carrier gas coming from a carrier source and through a make-up feeding line.

6.- A device according to claim 5, wherein said make-up line and said vaporization capillary column length outlet are connected in a way as to give rise to a rapid homogeneous mixing of the fed flows.

7.- A device according to claim 6, wherein a Venturi type fitting tube, having a reduced-diameter zone, houses, upstream said reduced diameter zone, a coaxial outlet of said vaporization capillary column length and the outlet of said make-up line, while downstream said reduced diameter zone, said fitting houses the inlet of said chromatographic column and the inlet of said splitting line.

8.- A device according to one of the preceding claims, and comprising also a cool trap at the inlet of said gas chromatographic column.

Fig. 1

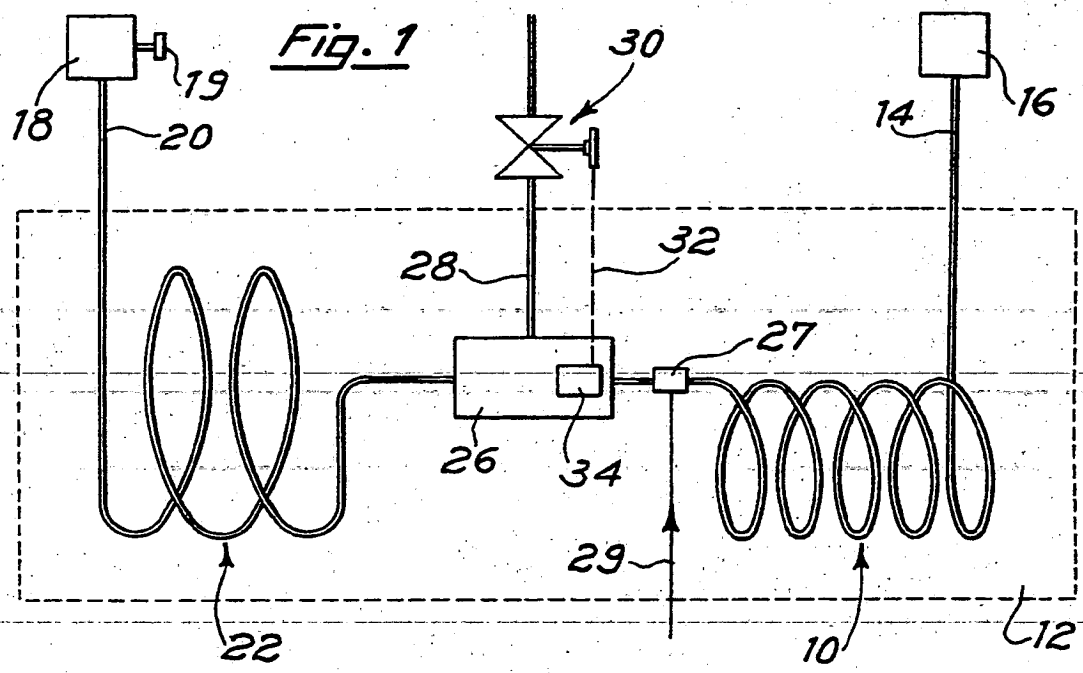

Fig. 2

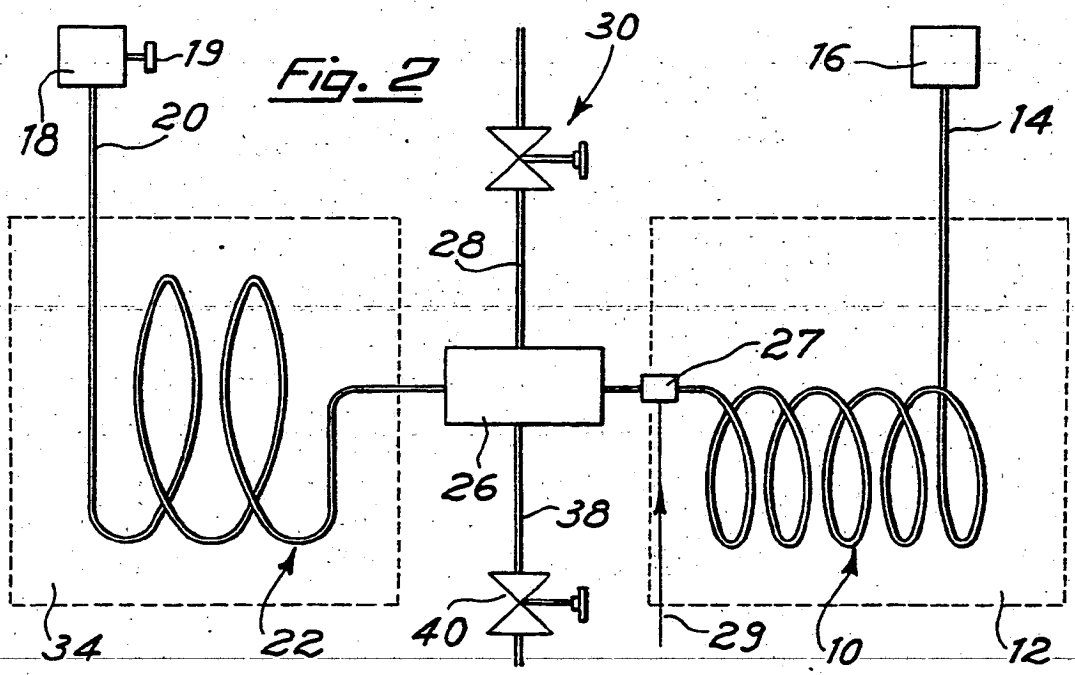

Fig. 3